

MAGRUDER NEWSLETTER

www.magruderchecksample.org

Winter 2022 Edition

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CHAIRMAN NOTES

There has been considerable activity in 2021 preparing for new sample schemes in the 2022 program. Labs can now choose from Regular, N, P, or K sample schemes. The Regular scheme includes one sample per month and covers a variety of fertilizers throughout the year as in past years. The N, P, and K schemes are being introduced in 2022 to cater to the needs of laboratories that support fertilizer manufacturers. With the introduction of these new sample schemes, several new labs have joined the program. The number of enrolled laboratories increased from 134 to 171. This issue of the Magruder Newsletter contains information on resources to help labs navigate data entry and interpret reports. It is my hope that both new and existing participants find this information useful.

The major goal of the Magruder program is to provide data and information to help labs maintain or improve the quality of results provided to their clients. There is a common saying that there is “strength in numbers”. This saying applies well to what the Magruder Proficiency Testing Program offers laboratories. Reviewing only the results of fertilizer testing that is conducted in your laboratory limits the ability to fully evaluate your laboratory’s performance. Greater assurance and satisfaction of your laboratory’s daily work is acquired from comparing your results with a larger number of laboratories that are analyzing the same sample. An article is presented here on the tools available in the Magruder program to help you have that strength in numbers. Additional resources on the following pages can help elevate laboratory performance including an article on control charting and opportunities (e.g., webinars) to learn more about instrument operation.

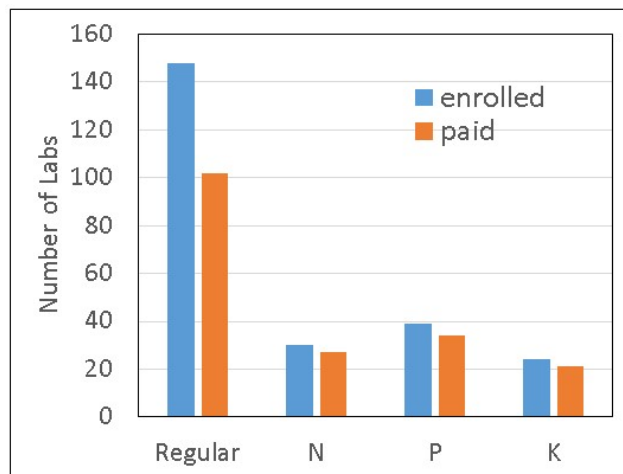
- *Frank Sikora*



PAYMENTS FOR 2022 ENROLLMENT

The Magruder program is adjusting to receiving payments prior to sending samples. Invoices for 2022 were initially sent out in August 2021 with a deadline of December 1, 2021 prior to the first sample of 2022 being sent out. A grace period of 90 days beyond December 1st has been given for labs to make payment before stopping sample shipments. The number of labs enrolled and paid in the Regular, N, P, and K schemes are shown in the Figure below. There are currently 51 unpaid labs. Samples will stop being shipped if payment is not made before the 90-day grace period.

The program has a new treasurer and a new method in which payments are received. The new treasurer is Matt Pearson and he can be reached at treasurer@aapfco.org. Payments currently are being made through QuickBooks on the web by credit card or bank transfers. Please contact Matt to make payment if your lab has not paid yet for 2022.



DATA ENTRY AND REPORTS

The Magruder website at www.magruderchecksample.org has instructional resources on submitting data and interpreting reports on the left-hand side of the website. The Lab Portal is where you go to enter data and view individualized lab reports. Entering the lab portal requires a user login and password. If an individual in your laboratory needs login credentials, contact Frank Sikora (fsikora@uky.edu). The “Instruction” link directs you to a page with a link to the Instruction Manual, which explains how to navigate the Lab Portal. Video instruction files are available on this same page.

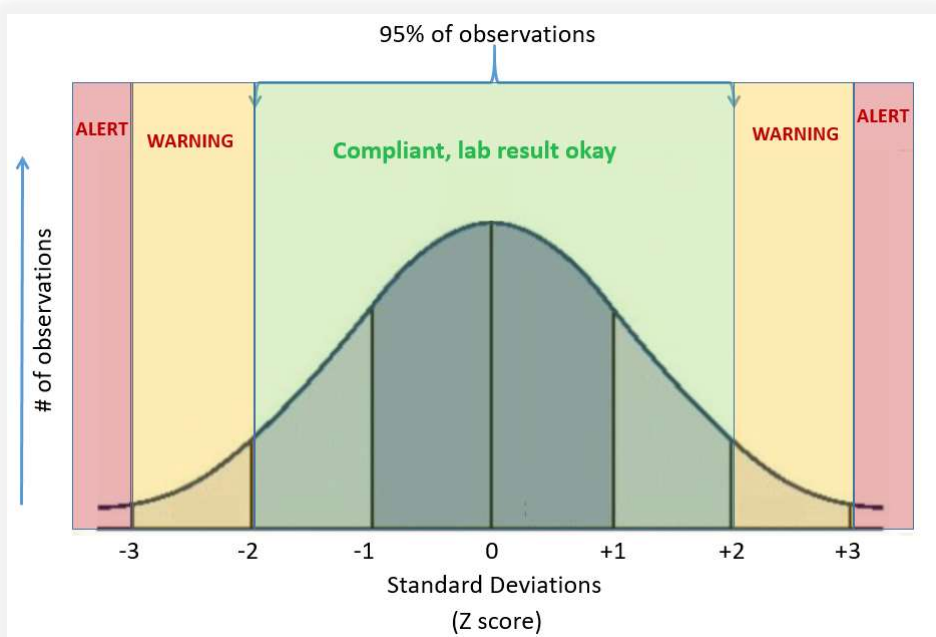
The “Method Codes” link opens the list of method codes that are used in the program. When reporting a laboratory result to the Magruder Lab Portal, the participant selects the method code that matches the method used to test a specific analyte. The method code has the format XXX.YY where the XXX identifies the analyte and the YY identifies the method used to determine the analyte. An example is method code 010.60. The first three numbers, 010, identify the analyte as Total Nitrogen. The last two numbers, 60, identify the method as combustion. If your laboratory uses a method that is currently not in the Method Code list, use method code “Other” with YY=99.

There is a lot of information provided on Magruder reports. Documents describing the various reports and explaining the data are found under “Reports Explained”. General reports with data from all participating laboratories can be found in the “Reports” link on the top green menu bar. Individualized reports for your lab can be found through the Lab Portal.

EVALUATING LABORATORY PERFORMANCE WITH MAGRUDER DATA

A primary goal of a proficiency testing program is to provide data and information for a laboratory to determine how well they are doing. The reputations of laboratories rely on their ability to provide correct results to their clients. In the absence of a proficiency testing program, it is difficult for a lab to know how correct their results are. The Magruder Proficiency Testing Program offers valuable tools to assess your lab's performance.

A Z-score is a value that lets you know where your lab result lies within the spread of data from all the other labs. A bell curve with a standard deviation is determined from the data submitted for an analyte. Robust statistics are used to avoid outliers from adversely exaggerating the standard deviation. Z-scores are determined for your lab considering all results with the same method in your Method Report Card and all results regardless of method in your Analyte Report Card. A Z-score of 0 would mean your lab result would be equal to the assigned value, which is the robust mean in the middle of the bell curve. A Z-score between -1 and +1 indicates your result was within one standard deviation from the robust mean while a Z-score between -2 and +2 is two standard deviations from the robust mean. Z-scores between -2 and +2 are colored green and are considered compliant. Z-scores between -3 and -2 or between +2 and +3 are further from the robust mean and are colored orange on the reports as a cautionary warning for the lab. Z-scores less than -3 or greater than +3 are colored red on the reports as an alert for labs to investigate why their result is so different from the other labs. Z-scores that are grey on the reports should be interpreted with caution. Grey indicates that the number of reported results (i.e., fewer than six) is too low for a good estimate of the robust mean and standard deviation.



You can download an excel file to track your Z-scores over time by clicking on “Control Charts” under “Samples, Reports & Data” at www.magruderchecksample.org. If prompted to enable macros and editing when opening this file, please enable these settings. Follow instructions on the StartUp sheet. Click on the ControlCharts sheet to view a chart of your Z-scores. You click on the various method codes listed in a box on the left side of the sheet to view Z-scores for different analytes. The chart on the left displays your Z-scores considering all methods

(from Analyte Report Card). The chart on the right displays your Z-scores considering all results for the method you used (from Method Report Card). A table of data under the charts is provided showing Z-scores, number of labs reporting, robust mean and robust standard deviation.

A Z-score evaluates how well your result lies within the spread of data from all other labs. The Z-score interpretations described above do not evaluate how close your value is to the “real” concentration. To determine the trueness of your result, a determination has to be made on where the true concentration lies for the sample. With 16 or more labs reporting a value for a particular analyte, a good estimate can be made on where the true value lies. This estimate is provided in the certificate of analysis (COA) for each sample and can be downloaded in the report tables at www.magruderchecksample.org by clicking on “COA”. The estimate is presented as a value with \pm standard uncertainty. Multiplying the standard uncertainty by 2 provides a range

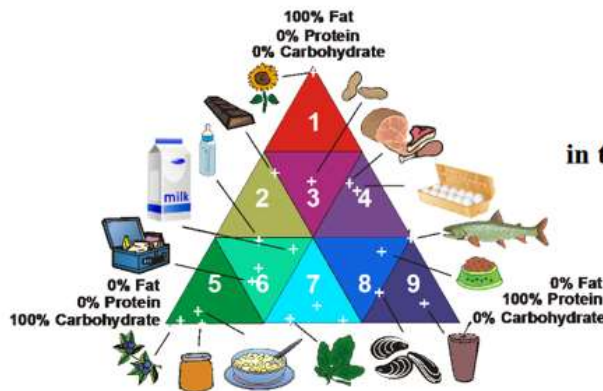
where the true concentration is expected to lie at a 95% probability level. For example, the true value for total N in sample 210211 is expected to lie somewhere between 21.60 and 21.84 at a 95% probability level.

If your laboratory result falls within the 95% probability range, you can be assured that your lab result is true. If your result falls outside of the 95% probability range but is close to that range, your result may still be true depending on your lab's measurement uncertainty. Laboratory results significantly outside of the 95% probability range are considered to be biased. A good reference for determining your lab's precision and trueness is shown below.

Free publication for assessing your laboratories precision and trueness from reference materials

Focus is on foods and dietary supplements but same principles can be applied to fertilizers

Available at <http://dx.doi.org/10.6028/NIST.SP.260-181>



NIST Special Publication 260-181

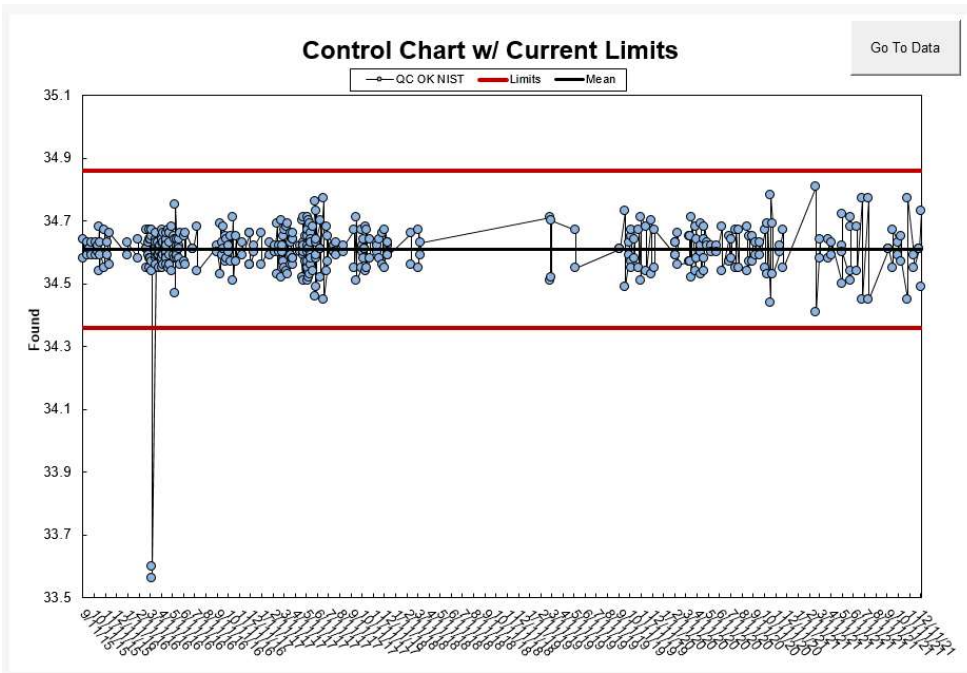
The ABCs of Using
Standard Reference Materials
in the Analysis of Foods and Dietary Supplements:
A Practical Guide

NIST
National Institute of
Standards and Technology
U.S. Department of Commerce

- Frank Sikora

USING CONTROL CHARTS IN QUALITY ASSURANCE

Every result obtained in an analytical laboratory must be defensible. There are several tools we can use to ensure the result is correct, unbiased, and defensible. In our last newsletter, we talked about one tool in our quality toolbox, quality reference materials. In this newsletter, let us think about a different tool from our quality toolbox, quality control charts. A control chart is very useful for visualizing trends and is easily evaluated for verification that a set of analytical data is acceptable. In order to maximize benefits from a control chart it should be reviewed each time a data point is added to it. Constructing control charts long after the analysis date only demonstrates historical control and is of minimal value for determining immediate run or batch acceptability. Control charts may include the following information: laboratory name, analyte, method of analysis, date(s), concentration range and analyst(s). Control charts can be constructed by hand, commercial QC or statistical software program, or spreadsheet.

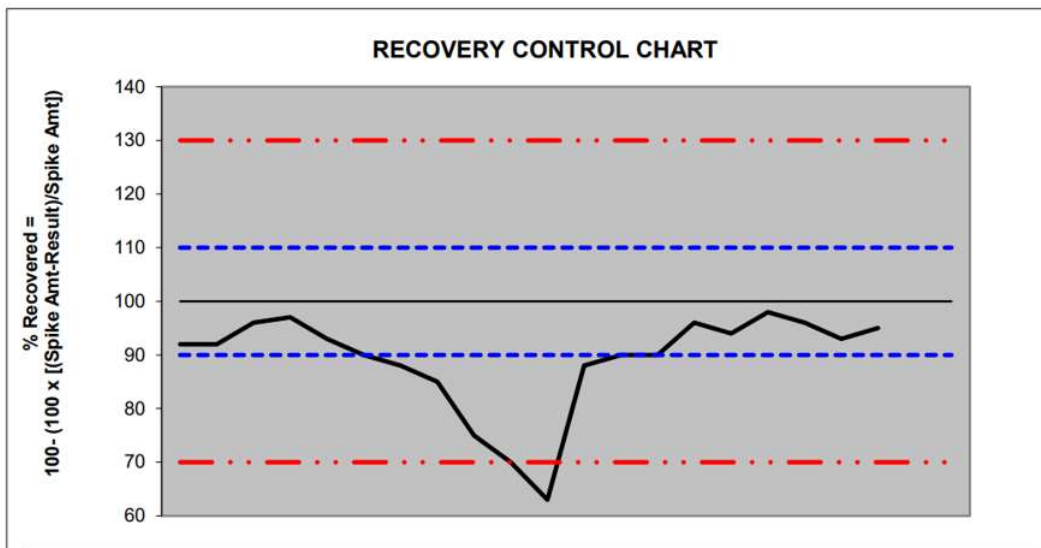


There is a wide variety of control charts from which to choose depending on what information you are seeking. An x-chart is a popular control chart for laboratories. A reference material or control sample is analyzed with each batch of samples. Once data have been collected for about 15 to 20 data points, the mean and standard deviation of the mean are calculated, and control limits are determined.

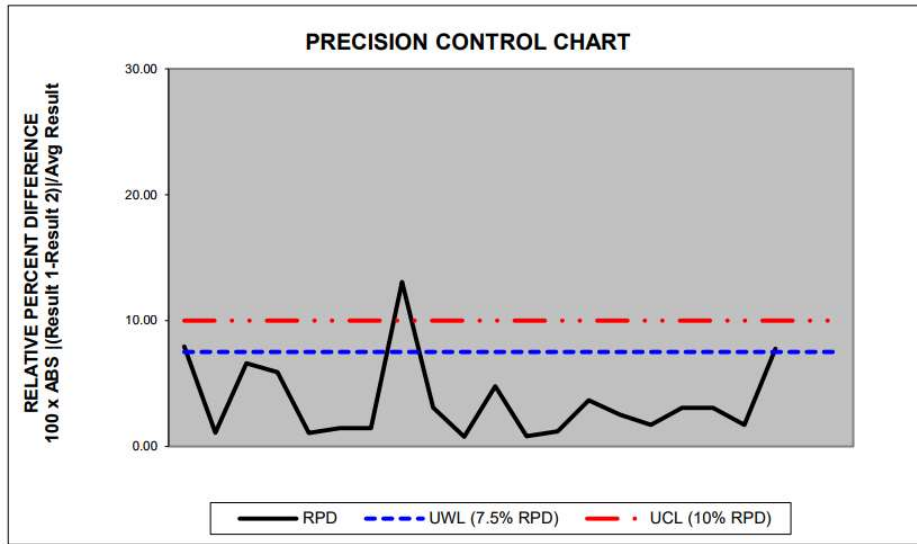
Consider the example control chart provided here. The bold black line is the mean. The bold red lines are 2.5 standard

deviations from the mean. In this control chart, any data point above the top line or below the bottom line indicates the result was out of control.

A control chart for matrix spike recovery of an analyte is shown below. Analyte with a known concentration in a reference material is added to the sample ideally with the sample matrix free of the analyte. The percent recovery of the analyte is determined on several occasions and can be plotted in a fashion similar to the chart shown below. The central line is 100% where perfect recovery is achieved. Control limits for recovery depend on the concentration of the analyte with the limits being tighter as the analyte concentration increases. The limits in the chart below are examples of limits for an analyte concentration of 10 ug/kg (ppb) with limits of 70 and 130% and an analyte concentration of 0.1% with limits of 90 and 110%.



An R-chart is useful for monitoring repeatability. This is the precision of duplicate samples analyzed in the same batch of samples. The range is then calculated for each pair of duplicates. The range or relative percent difference can be plotted for each batch of samples analyzed. The graph below shows an example of relative percent difference. Control limits can be set based on expected repeatability.



Each laboratory should have a plan on what to do when a control chart shows out of control data. At the University of Kentucky Division of Regulatory Services, our supervisors will not pass a set of samples if the result from the quality reference material was beyond the control limits of a x-chart.

Information is available in control charts that can be used to determine if something is awry with your method. Ideally, data from a control chart shows a random pattern. When the data does not follow a random pattern, follow-up investigation may be required on the method. A guideline for nonrandom patterns to look out for in control charts is provided below.

Table 1. Proposed Rules for Interpreting Control Charts Out-of-Control Data¹
Any point beyond the control limits
Seven (or more) consecutive points above or below the centerline
Seven consecutive increasing or decreasing points
Two out of three beyond two sigma on the same side of the centerline
Four out of five beyond one sigma on the same side of the centerline
Ten out of 11 consecutive points on the same side of the centerline
Twelve out of 14 consecutive points on the same of the centerline
A series of points "hugging" the centerline
A series of points "hugging" the control limits
Fourteen consecutive points alternating up and down (saw-tooth pattern)
Any nonrandom pattern (e.g. A cycle)

¹from Quality Progress, November 1994, "Are You Getting the Most Out of Your Control Charts?"

Using control charts as just described is an integral component of a quality control operating procedure. Using control charts and the principles described can help labs defend their data and show their customers the laboratory is in control of the data it generates.

VENDOR'S CORNER

We are adding a recurring feature to the newsletter. Vendor's Corner is a place to discover resources, such as training opportunities, that could be helpful to your laboratory team. Agilent Technologies, Inc. has graciously provided information for this introductory Vendor's Corner. Ideas for future Corner articles are welcomed!

- Sally Flowers

Knowing the right information at the right time can help you cut through the everyday complexities of your samples, instrument needs, regulations, other demands on your time and resources – and help you handle unexpected challenges. The following resource HUB can provide information on every technique for most applications. Regardless of the instrument, the method or consumable used, tips and tricks can help ensure you get the right answer the first time.

1. Training & Events - Stay connected with the latest application news, as well as software enhancements by registering for a live or pre-recorded event. These technical events are a convenient way to keep your knowledge up to date on the latest technologies and learn from industry professionals.
- <https://www.agilent.com/en/training-events>
 - a. Atomic Spectroscopy Webinar Curriculum – The Atomic Spectroscopy/Atomic Spectrometry webinar series can help any analyst stay up to date with emerging trends and applications in ICP-QQQ, ICP-MS, ICP-OES, MP-AES, and AA, as well as tips and tricks for improved interference removal, maintenance and method optimization for atomic applications, regardless of instrument brand.
- <https://www.agilent.com/en/training-events/eseminars/atomic-spectroscopy>
 - b. Atomic & Molecular Spectroscopy Digital Workshops & Bootcamps -The Bootcamp and Workshop series provide in-depth information and insights on hardware, software, and application per specific method criteria.
- <https://www.agilent.com/en/training-events/eseminars/spectroscopy-workshops-bootcamps>
 - c. Molecular Spectroscopy Webinar Curriculum - The Molecular Spectroscopy/Atomic Spectrometry webinar series can help any analyst stay up to date with emerging trends and applications for UV-Vis, UV-Vis-NIR, FTIR, Imaging, Fluorescence, and Raman techniques as well as tips and tricks for improved workflows, maintenance and method optimization for applications, regardless of instrument brand.
- <https://www.agilent.com/en/training-events/eseminars/molecular-spectroscopy>
 - d. LC & LC/MS Column Webinars – Column selection and chemistries can be confusing. The LC and LC/MS webinars can clarify the chemistry and streamline method optimization.
- <https://www.agilent.com/en/training-events/eseminars/lc-lc-ms-column-e-seminars>
 - e. Chromatography and Mass Spec Educational Webinar Series – Agilent Application Scientists will cover a number of different chromatography top showcasing application-specific workflows to enhance your analytical needs.
- <https://www.agilent.com/en/training-events/eseminars/832>

2. Agilent Education & Training – On-line theory and method development training modules for all techniques
- <https://www.agilent.com/en/training-events/events/agilent-education>
3. ICP-MS secrets - watch the videos – Secret tips and tricks for ICP-MS applications
- <https://explore.agilent.com/7850-icp-ms-tips-videos>
4. Agilent ICP-OES video tips – Secret tips and tricks for ICP-OES applications
- <https://explore.agilent.com/icpoes-video-tips>
5. Agilent Atomic Spectroscopy Resource Hub – Maintenance guidance for AA, ICP-OES and ICP-MS instruments
- <https://explore.agilent.com/spectro-resource-hub>
6. Cannabis & Hemp Testing and Analysis – Up to date method guidance for cannabis and hemp testing laboratories.
- <https://www.agilent.com/en/solutions/cannabis-hemp-testing>

- *Courtesy of Christine Rivera, Agilent Technologies, Inc.*

UPCOMING MEETINGS:

TFI Annual Business Conference (January 31-February 2; Orlando, FL)

Visit <https://www.tfi.org/content/annual-business-conference>

2022 AAPFCO Winter Annual Meeting/Methods Forum (February 13-18; Mobile, AL)

Visit <https://www.aapfco.org/meetings.html>

Fluid Fertilizer Foundation – Fluid Forum (February 21-22, 2022; Scottsdale, AZ)

Contact info@FluidFertilizer.org

2022 Commodity Classic (March 9-12; New Orleans, LA)

Visit <https://commodityclassic.com/for-media/convention-news/article/2021/04/2022-commodity-classic-show-floor-opens-for-exhibitors>

Southwestern Fertilizer Conference (July 10-14; Nashville, TN)

Visit <https://www.swfertilizer.org/>

TFI: InfoAg Conference (July 25-27; St. Louis, MO)

Visit <https://infoag.org/>

2022 AAPFCO Summer Annual Meeting (July 31 - August 3; St. Louis, MO)

Visit <https://www.aapfco.org/meetings.html>

MAGRUDER COMMITTEE ROSTER

Name	Organization
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Sally Flowers, Newsletter Editor	Kansas Department of Agriculture, Manhattan, KS
Ametra Berry	Georgia Department of Agriculture, Atlanta, GA
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